FEDERAL REPUBLIC OF GERMANY

GERMAN PATENT OFFICE

Patent disclosure

DE 32 33 557 A1 B 01 J 19/18

(withdrawn on 18.10.84 no foreign country counterparts)

File reference: P 32 33 557.1

Date of application: 10.9.82

Date of disclosure: 15.3.84

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Reactor with pipe scraper pigs

Build-up of deposits can be prevented on the internal tubular wall of a reactor if one or more pipe scraper pigs (4, 5) are circulated with the liquid flow during the reaction. (32 33 557).

Patent claims

1 Method for carrying out continuous or sequential reactions, in liquid phase, wherein the reacting mixture is moved through a tubular reactor, characterised by the fact that during the reaction in the tubular reactor one or more scraper pigs are circulated with the liquid flow which prevents deposits forming on the internal wall of the reactor.

2 Method for carrying out photo-reactions in accordance with claim 1, characterised by the fact that the reacting mixture is irrradiated from the exterior in the tubular reactor.

3 Tubular reactor for carrying out continuous or sequential reactions, preferably photo-reactions, in liquid phase, including a reaction tube (1), incoming and outgoing product valves and pump (15) to move the liquid in the reaction tube, characterised by the fact that one or more scraper pigs (4, 5) are circulated with the reacting liquid in the reaction tube.

4 Apparatus in accordance with claim 3, characterised by the fact that at least one scraper pig is present, the reaction tube is designed as a loop (1) through which the liquid always flows in the same direction and there is at

least one scraper pig trap (2, 3) in the tube loop (1) between incoming (6, 9) and outgoing product valves (7, 10) on the tube.

5 Apparatus in accordance with claim 3, characterised by the fact that the reacting mixture circulates through two reaction tubes (30, 31) of the same length and same volume, each provided with a scraper pig (32, 33), first in one tube then the other, whereby each tube is cleaned alternately in both directions.

6 Apparatus in accordance with claim 3, characterised by the fact that one or more spherical-shaped scraper pigs (53, 54, 61) are propelled with the reacting liquid by a pump (52) into the reaction tube (56), separated from the liquid behind the reaction tube (60) and returned to the pump (52) by gravity.

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Reactor with pipe scraper pigs

The invention concerns a method and apparatus for carrying out continuous or sequential reactions, in liquid phase, wherein the reacting mixture is pumped through a tubular reactor.

Tubular reactors for carrying out continuous chemical reactions are known. Their use is however questionable if deposits from the reaction products form on the internal wall of the reactor which is frequently the case with polymerisation and photo-reaction processes. On account of the unavoidable interruptions in operation for cleaning purposes in spite of various experiments, for example photo-initiated emulsion polymerisation of water-soluble monomers is not carried out in accordance with DE-OS 2 354 006 in such a continuous reactor. Apparatus as described in DE-OS's 956 542, 2 009 748, 2 050 988, 2 523 587 and 3 008 660 and EP 0 036 819 is used to a greater extent; but it is evident even at first sight that such complicated apparatus has to be more sophisticated and liable to disruption.

The object of the invention in connection with reactions in which disruptive deposits form is to propose a method and specify apparatus which will prevent such deposits forming on the internal wall of the reactor and which will allow longer periods of non-interrupted operation.

The object is achieved by the fact that during the reaction in the tubular reactor, one or more pipe scraper pigs are circulated with the liquid flow which prevents deposits forming on the internal wall of the tube. Special versions are described in the sub-claims.

Pipe scraper pigs are known in pipeline construction and are used for cleaning, draining, separating batches, conducting hydrostatic pressure tests etc. However it has not been realised up to now that deposits can also be removed from tubular reactors with such pipe scraper pigs. This may be linked with the observation that, when cleaning after closing down a reactor, in most cases a difficult-to-remove crust of hard or rubbery consistency is found, which cannot be eliminated with the aid of a pipe scraper pig.

Because with this invention initial deposits are removed shortly after they form, no hard or rubbery tightlyadhering deposits at all build up.

Pipe scraper pigs which can be used in accordance with the invention are for example described in German Patents 28 01 378 and 30 32 532. Tube diameters of the reactor are normally between 25 and 800 mm. Pipe scraper pigs as well as the corresponding trap devices are commercially available for these dimensions. The pipe scraper pig is circulated by corresponding pressure of the liquid pushing against it. It remains so tight against the internal tubing wall that the reacting liquid stays clearly separate as a result of the pipe scraper pig.

The pipe scraper pigs themselves consist of soft or semi-hard natural or plastic material, preferably rubber or polyurethane. Also scraper pigs with flexible metal parts or metal brushes as well as combinations of metal and plastic can be used. Of the numerous possible shapes, spheres, cylinders, cylinders with rounded edges as well as cylindrical bodies with thick lips and thick strips on the outer circumference are preferred. Hardness, dimensions, measurements compared to the tube-circuit as well as shape and curvature of the scraper pig are governed amongst other things by the type of deposit and the tolerances and radii of the tubes. In the case of glass tubes permissible pressure loss must also be considered as a design criterion which is determined by the strength requirements of the tube. The number of scraper pigs used is determined by the speed of deposit formation and cross-linking and hardening of the deposits formed as well as by permissible pressure loss.

The apparatus according to the invention is illustrated in the drawing and the method is described in detail below. The following illustrations are provided:

Fig 1	Reactor with a tube loop
Fig 2	Reactor made from two tubes working in unison
Fig 3	Tubular reactor with no valve change-over.

The tubular reactor in Fig 1 consists of a closed tube loop 1 which is interrupted by ball valves 2, 3. Ball valves 2 and 3 have the same aperture cross-section as tube loop 1 and allow scraper pigs 4 and 5 to pass through. In this special example scraper pig 4 is introduced exactly into the lock between the two ball valves while scraper pig 5 is just meandering through tube loop 1. The outgoing solution is taken through valve 6 and the reaction product is extracted through valves 7 and 8. Ball valve 2 is closed. By closing valves 3, 6 and 7 and opening valves 2, 9 and 10, the outgoing solution which is pumped in through valve 9, starts to move scraper pig 4 again whereby the reaction product is discharged through valve 10.

As soon as the scraper pig has passed the inlet at valve 6, changeover to the old valve position (valves 3, 6 and 7 open, valves 2, 9 and 10 closed) is possible and the trap between ball valves 2 and 3 is ready to accept the other pipe scraper pig 5 coming round. After changing over to the old valve position a mixture of outgoing solution and reaction product which can be separated from the reaction product through tube 14 and returned via pump 15 by opening valve 11 and closing valve 8 runs for a short time through valve 7. The reaction product is drawn out through tube 16.

The valve changeovers required at the end of each scraper pig run are preferably activated automatically. The control impulse required is triggered off here by the increase in pressure when the scraper pig hits the closed ball valve 2 or another suitable device. If the trap between ball valves 2 and 3 consists of a glass tube, the control impulse is preferably emitted by a photocell 17.

Another version of the tubular reactor according to the invention is shown in Fig 2. It is made up of two identical straight tubes 30, 31 both containing a scraper pig 32, 33. Here no ball valves are needed in the reactor as the scraper pigs in the tubes with the same diameter and same volume move continuously backwards and forwards. The scraper pigs have the general shape of the tube end so that the space between scraper pig and tube end can empty as much as possible. The three phases described below are typical for this apparatus:

1. Pumping into the lower reactor: the reaction solution is taken through tube 34 to lower reactor tube 30 into which it enters via tubes 35 and 36 and threeway valves 37 and 38 alternating at both ends. When it is pumped in through tube 35 scraper pig 32 moves from left to right.

2. Transfer into the second reactor: as soon as scraper pig 32 arrives at the right end of the tube the incoming flow changes direction and therefore alters the direction of the scraper pig by changing valve 37 into the through flow position and by opening valve 38 in the direction of tube 36. Therefore the contents of lower tube 30 are pumped through tube 39 into upper reaction tube 31 whereby scraper pig 33 is propelled in front of the liquid.

3. Discharge of the reaction product: when scraper pigs 32 and 33 have arrived at the left or right end of the tube, the incoming flow is again reversed and taken via valve 37. At the same time valve 38 is changed into the

through flow position so that the liquid enters upper tube 31 via tube 40, as a result scraper pig 33 is again pushed to the left and the reaction product is discharged via tube 39 and open valve 41 into outlet 42.

All incoming products principally pass through these three stages whereby product coming through tube 35 is pumped via tube 39 into the upper reactor and discharged via valve 41 and vice versa.

By reversing the direction of flow in the reactor with ideal piston-type circulation without re-mixing, uniform retention time for all constituents is achieved.

Also with this apparatus valves are preferably automatically activated whereby valves 37, 38, 41 and 42 are changed at the same time into another direction. The control impulse can, as in the case of the apparatus shown in Fig 1, be provided through the increase in pressure when the scraper pigs hit the tube ends or other suitable devices, in the case of glass tubes preferably through a photocell.

Another tubular reactor according to the invention is shown in Fig 3. The apparatus is especially suitable for large flowrates with less retention time. The reaction solution passes through valve 51 to the suction inlet of pump 52, which at the same time moves spherical-shaped scraper pigs 53, 54 and 61. For example a rotary pump with set-back rotor is suitable in this case. The capacity of the pump is regulated by return flow valve 55. After passing through tubular reactor 56 with rising tube 57 and outlet elbow 58, product and tube scraper pig enter vessel 59. As a result of a slanted rough mesh grid 60 scraper pigs 61 are separated and then run by gravity through tube 62, tube bend 63 and conical tube 64 to the suction inlet of pump 52. A high S.G. of the tube scraper pig is necessary for good suction which for example is achieved by inserting a metal core. The reaction product flows out through tube 65 from separation vessel 59.

Figs 1 and 2 show cooling mantles 70 and 71, 72 in which a cooling fluid enters at 73 and 74, 75 and flows out at 76 and 77, 78. All types of cooling are possible; with smaller tube dimensions mantle cooling is preferable, in the case of larger dimensions trickle film cooling is preferable. Also cooling with gas is feasible.

For photo-reactions, reactors are made from glass tubes. Figs 1 and 2 show the necessary lamps with 80, 81, 82, the reflectors with 84, 85, 86.

All sources of radiation whose emission is in the range of between 1500 and 5000 A, preferably 3000 to 4000 A can be used.

Sunlight is also emitted in these wavelengths. Mercury vapour, xenon, wolfram and carbon arc lamps, especially tungsten tubes are advantageous. Water is preferred for the cooling fluid as it does not absorb light in the wavelength range quoted.

The advantage of the apparatus shown in the examples lies in the fact that the reactors have no mechanical components on which deposits can form and that the simple working principle keeps the entire internal surface of the reactor free from deposits without high structural expense.

From a technical point of view in regard to reaction processes there is also the advantage the circulating scraper pigs produce a piston-type circulation and therefore provide the optimum retention time distribution of the ideal tubular reactor. This advantage is important for reactions which are intended to achieve a high degree of conversion. A typical example is photo-polymerisation of acrylamide.

Example:

A loop reactor in accordance with Fig 1 had the following dimensions:

Glass tube: ϕ DN 50, loop length 4.74 m, useful content 9.3 litres, no cooling.

Scraper pig: cylindrical shape, rounded edges R = 20 mm, diameter 51.2 mm, length 80 mm, material: polyurethane, foamed; 1 scraper pig.

Lamps: ten Philips tubes TL 20 W/09.

Pump: eccentric worm pump.

It is known with photo-initiated polymerisation of acrylamide/acrylic acid in oil emulsion that deposits quickly form in all reactors, consisting firstly of a sticky film which changes after a time as a result of cross-linking into a solid or rubbery layer which can only be removed with difficulty by mechanical means or by solvents.

In the reactor according to the invention the glass surface remained deposit-free in continuous operation (flowrate 9 litres per hour) during the whole trial period of 12 hours. It was seen that the circulating scraper pig immediately removed any initial deposits.

The emulsion product was formulated as follows:

Water phase:

7.50 kg acrylamide
1.78 kg acrylic acid
7.50 kg water
2.51 kg 50% caustic soda and
2.25 g 4.5 diphenyl 3 keto 4 ethoxy-sodium valeriat as photo initiator

Oil phase:

paraffin mixture
as emulsifier
ethylenoxide as emulgator

Number: 32 33 557
Int Cl^3: B 01 J 19/18
Date of application: 10th September 1982
Date of disclosure: 15th March 1984

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	840614 +	RECEIPT OF EXAMINATION APPLICATIONS PAR 44
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Nummer: 32 33 557
Int. Cl.3: B 01 J 19/18
Anmeldetag: 10 September 1982
Offenlegungstag: 15 März 1984

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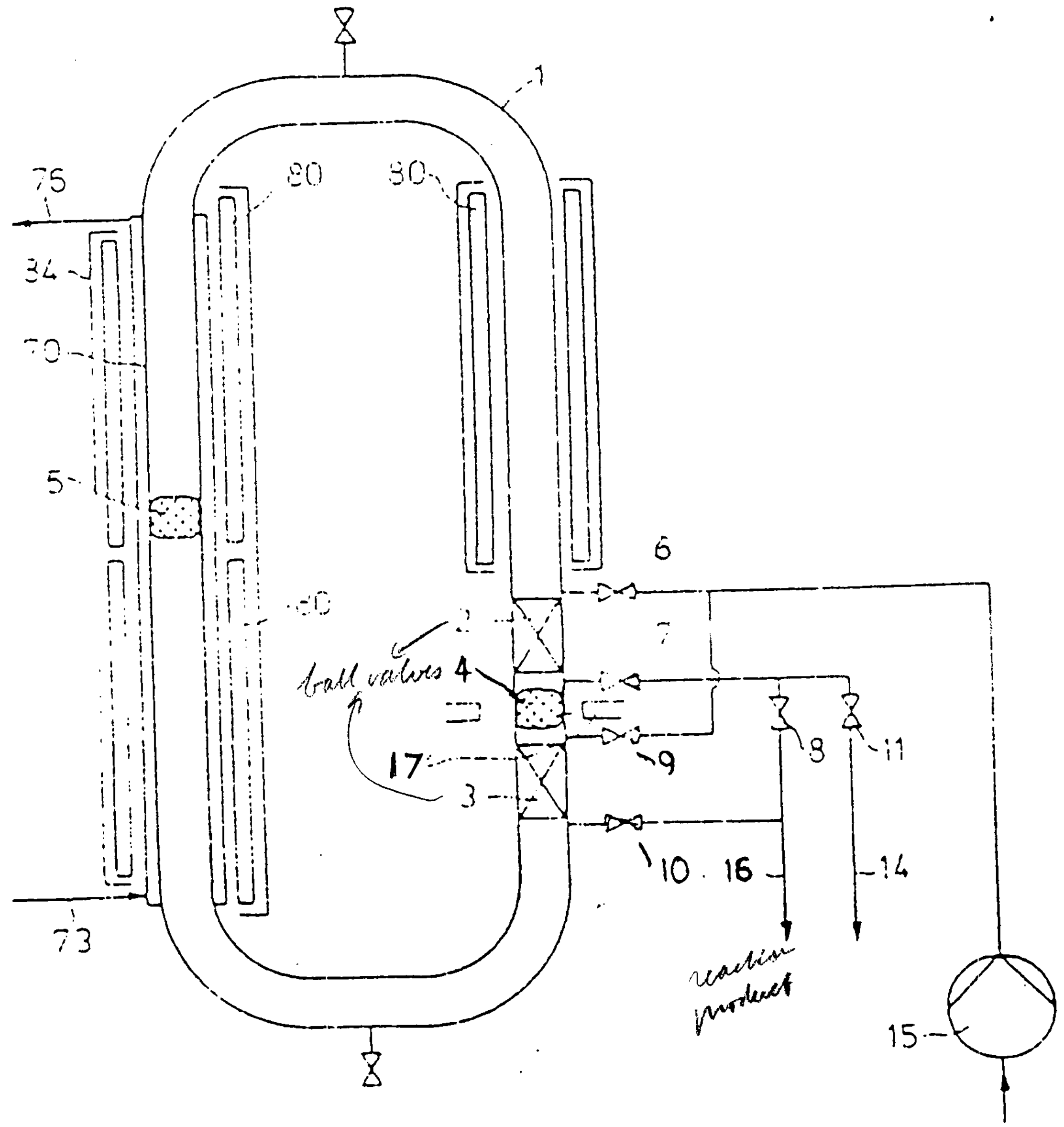

FIG. 1

2.2

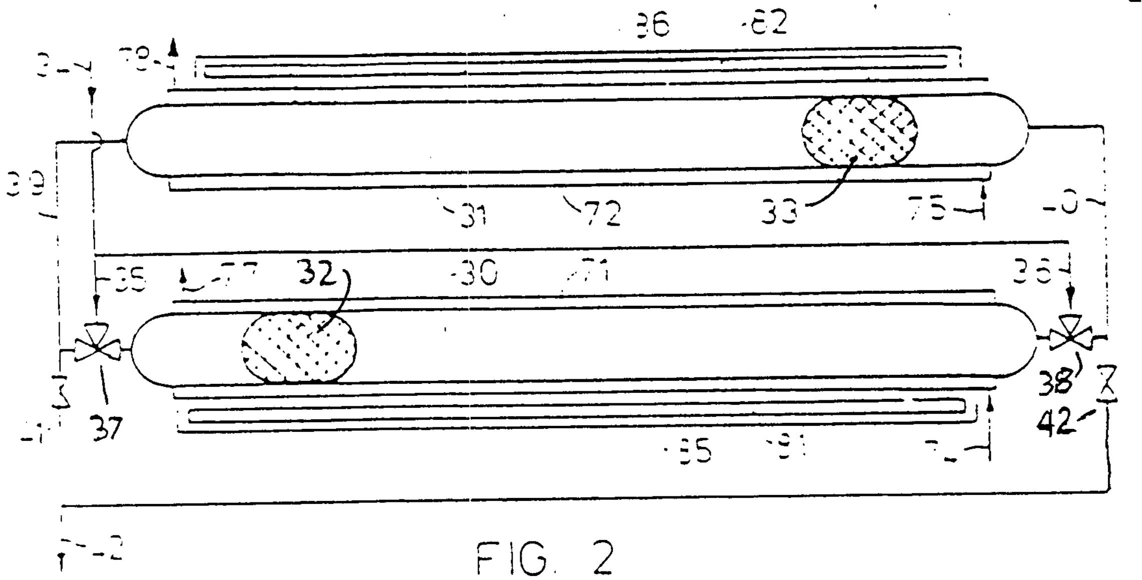
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FIG. 2

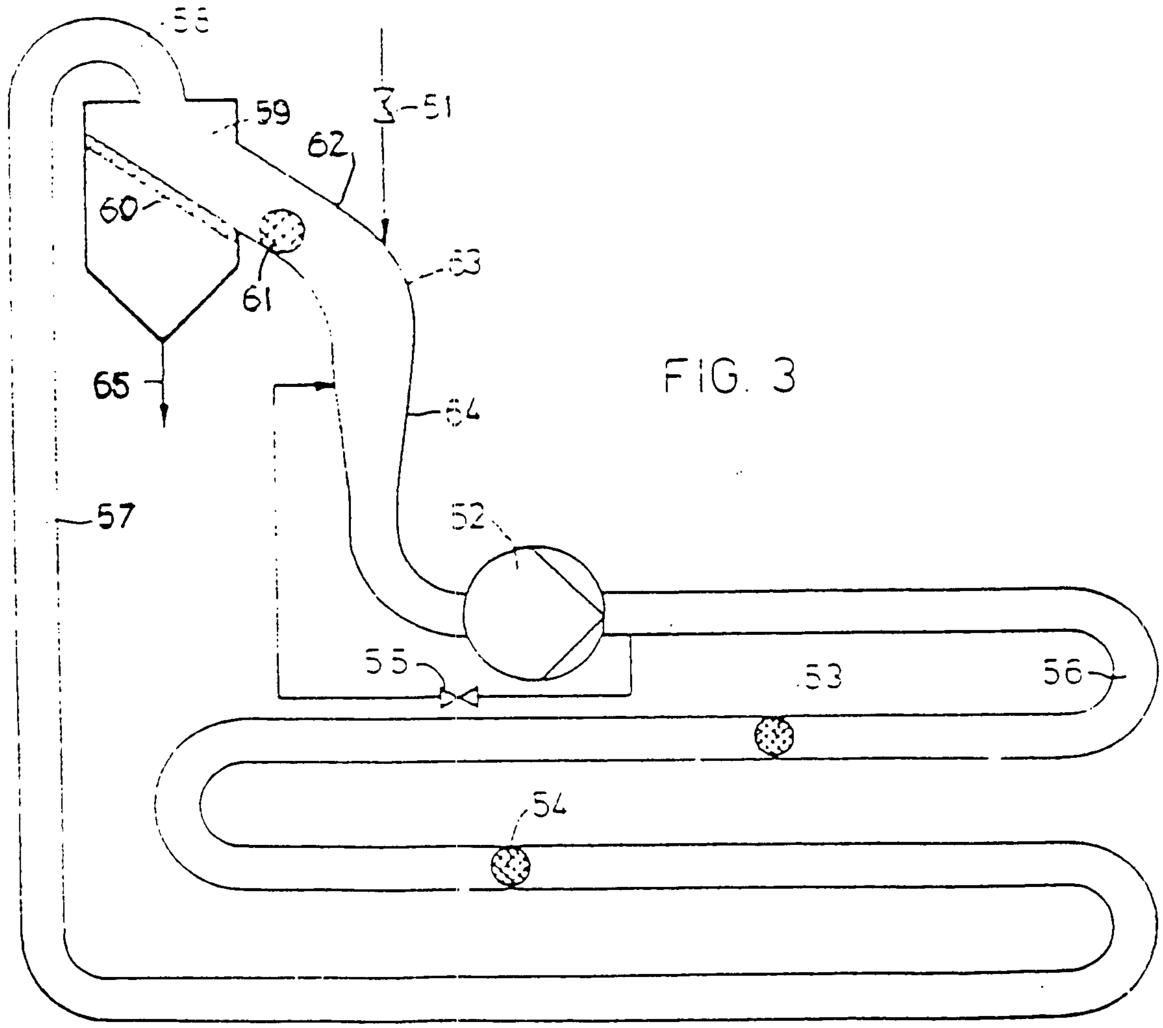
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FIG. 3